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A study of retention of sugars in the process of clarification of pineapple juice (*Ananas comosus*, L. Merril) by micro- and ultra-filtration

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Abstract

The aim of this work was to evaluate the loss of sugars (glucose, fructose and sucrose) in pineapple juice (*Ananas comosus*, L. Merril), Pérola cv., hydrolyzed with commercial pectinase (Ultrazym 100G) alone and combined with a cellulase (Celluclast) as a pre-treatment, and after clarification by cross flow micro- and ultra-filtration, using two different module geometries (plate/frame and tubular systems) to select the membrane process that would best preserve these nutrients. Membranes of polysulfone (PS), polyethersulfone (PES) and polyvinylidene fluoride (PVdF) to micro- and ultra-filtration were used. The membrane pore diameters and cut-offs were: 0.1, 0.45 μm, and 50, 100 KDa (PS), and 0.3 μm and 30–80 KDa (PES and PVdF). All processes were operated at different trans-membrane pressures (TMP), at room temperature (25 °C ± 2). The sugar contents of the clarified pineapple juices determined by HPLC revealed significant differences at a 5% level. These results showed that the membrane pore diameters or cut-offs as well as the module geometry influenced the clarified juice sugar contents. It was observed that the sugar content was more reduced when the 30–80 KDa tubular membrane at 1.5 bar was used for pineapple juice clarification. Although the best total sugar recoveries have been observed in juices clarified with polysulfone membranes (50 KDa – 7.5 bar), the use of 0.3 μm PES, due to its tubular configuration and module geometry, is more attractive and appropriate.

Keywords: High performance liquid chromatography; Sugars; Pineapple juice; Micro-filtration; Ultra-filtration

1. Introduction

The contents of sugars in fruit juices, combined with their acidity and aromatic profile, promotes the exotic and characteristic flavors of each fruit (Carvalho et al., 2003). Pineapple juice (*Ananas comosus*, L. Merril) is largely consumed around the world, mostly as a canning industry by-product, in the form of single strength, reconstituted or concentrated, and in the blend composition to obtain new flavors in beverages and other products. Total pineapple production worldwide is nearly 16 million tons with 12 countries accounting for 80% of the total pineapple production. Three-quarters of the 7.4 million tons of pineapple traded in the world are canned or in the form of juice. In 2004, two-thirds of the pineapple exported consisted of juice (single or concentrated), and the remaining one third was canned (rings, slices or cubes). Canned pineapple and pineapple juice market has increased fourfold worldwide since 1984 from 1.3 to 5.6 million tons (fresh fruit

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equivalent). The processed pineapple market is currently dominated by Asia, mainly Thailand, The Philippines, and Indonesia, and, more recently, Vietnam (Vagneron et al., 2005). The Brazilian pineapple production was, approximately, 325 million fruit only in the state of Paraiba, its major producer, accounting for over US $ 80 million. Brazil is considered the main pineapple producing country in the world since 2005, with over two billion tons ranging from 2,145.027,000 to 2,292.470,000 (IBGE, 2007; EMBRAPA, 2007). In 2005, pineapple was exported to 15 countries, most located in the European Union. Pineapple juice is the main pineapple product exported and Holland is its major importer (EMBRAPA, 2006). The Pérola variety studied in this work has high sugar content (14–16 °Brix), and low acidity and it is mainly consumed *in natura* or used for juice production (single strength or concentrate). Smooth cayenne is the most cultivated variety worldwide with high sugar content (13–19 °Brix), and high acidity, compared with other varieties. These characteristics make both varieties appropriate for industrialization (first in industrial importance) and exportation for *in natura* consumption. Singapore Spanish ranks second in industrial importance, being largely cultivated in Malaysia, with low sugar content (10–12 °Brix), and low acidity. Other pineapple varieties cultivated around the world are: the Queen, largely cultivated in South Africa and Australia, rich in sugar content (14–16 °Brix), low acidity, and long post-harvest life; Red Spanish, also with low sugar content (10–12 °Brix) and low acidity but very aromatic, and Perolera or Branco de Pernambuco, cultivated in Colombia, Brazil and Venezuela, with sugar content around 13 °Brix, low acidity and high ascorbic acid content.

More recently, the consumer market has been equally receptive to limpid fruit juice and those obtained by the traditional clarification processes using gelatin, bentonite (Wosiacki et al., 1992; Stocké, 1998; Youn et al., 2004; Tajchakavit et al., 2001) as well as by processes using UF and MF membranes.

On the other hand, other finning agents, such as polyvinylpolypyrrolidone, activated carbon, and enzymes previously to membrane clarification processes have been recently investigated (Youn et al., 2004 and Rai et al., 2007). According to several authors, UF and MF membranes can be used for clarification processes (Tallarico et al., 1998; Carneiro et al., 2002, 0.3 μm membrane; Youn et al., 2004, 0.1 μm membrane; Matta et al., 2004, 0.3 μm membrane; Cianci et al., 2005, 0.3 μm membrane; López et al., 2005, 0.45 μm membrane). Membrane processes use tangential filtration and are based on pressurized feed, parallel to the membrane surface permeating the solution, leading to a limpid product, and one retentate that re-circulates within the system. On the other hand, membrane technology such as MF and UF is very important for industrial purposes because of their high productivity and reduced cost, resulting in yield improvement and high quality product (Girardi and Fukumoto, 2000; Drioli and Romano, 2000; Youn et al., 2004). In 1994, non-hydrolyzed pineapple juice from Pérola variety clarified by UF and MF was reported to produce a pineapple soft drink in Brazil (Carvalho et al., 1998). Since then, several projects have been developed on the clarification of pineapple and other fruit juices to investigate their composition, color, cold sterilization (Güell and Davis, 1996; Carvalho et al., 2002b; Carneiro et al., 2002; Bruijin et al., 2002; López et al., 2005), consumer market, as well as process optimization and influence on the aromatic profile.

The importance of the MF/UF processes lies in the preservation of the natural fruit constituents, as well as the volatile aroma profile, improving the microbiological quality of the permeates on account of the great potential of the membranes for industrial application of limpid juices and beverages, whose presentation can be very attractive to consumers (Viquez et al., 1981; Tallarico et al., 1998; Carvalho et al., 2002a; Cassano et al., 2004; Vaillant et al., 2005).

The main advantage of the clarification processes using MF and UF membranes is that they can operate at room temperature, avoiding the deleterious effects of thermal treatment, such as sugar caramelling and browning. On the other hand, several authors recommend previous enzymatic hydrolysis over these processes in juices and fruit pulps to reduce viscosity, improve permeate flux, and reduce fouling (Hashizume and Lattimer, 1973/74; Wosiacki et al., 1992; Sreenath et al., 1994; Brasil et al., 1996; Cardoso et al., 1998; Sakho et al., 1998; Pelegrine et al., 2000).

Preservation of sugars, vitamins and other constituents as well as similarity with the original product are fundamental requirements for the viability of these processes and consumer acceptance. The membrane pore diameter, or cut-off material, configuration, and trans-membrane pressures applied can influence the higher or lower nutrient preservation in the permeate juice.

The aim of this work was to evaluate losses of sugars (glucose, fructose and sucrose) in hydrolyzed pineapple juice (*A. comosus*, L. Merril) before and after clarification by cross flow ultra- and micro-filtration using two different module geometries (tubular and plate and frame) to select the process that best preserves these nutrients.

2. Materials and methods

2.1. Pineapple juice

Pineapple juice (*A. comosus*, L. Merril) from the Perola variety was processed at CETEC – Food and Beverages in Vassouras, Rio de Janeiro, Brazil, from 2.0 tons according to the following steps: selection, cleaning, manual peeling without removing fruit core, cutting, pulping (pressing), fining, and storing at −20 °C (Fig. 1).

2.2. Enzymatic treatment optimization

To minimise fouling and reduce juice viscosity, assays were carried out on a laboratory scale with Ultrazym 100G enzyme isolatedly (EC 3.2.1.15, CAS n. 903275-1,

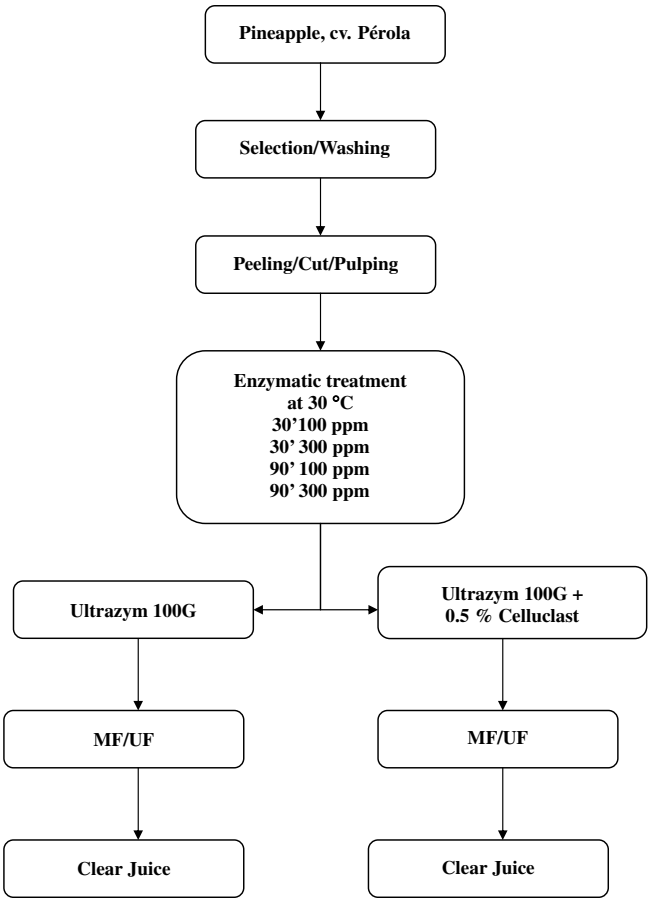

Fig. 1. Juice process and enzymatic treatment for membrane processes.

pectinase – IUB, 2007), and combined with 0.5% Celluclast (EC 3.2.1.4, CAS n. 90012-54-8, cellulase – IUB, 2007) with food grade (generally recognized as safe – GRAS), both from Novozymes, Kalundborg, Denmark (Table 1). Samples of 300 mL single strength juice were initially heated in a water-bath at 30 °C, followed by the addition of 100 and 300 ppm of Ultrazym 100G for 30 and 90 min, as well as 200 ppm for 75 min, under constant and controlled shaking at 100 rpm (±5) using a FANEM shaker. At the end of each assay, the enzyme was inactivated and the mixture heated at 85 °C for 5–10 min, cooled and stored at −20 °C. The same procedure was carried out using the Ultrazym 100G + Celluclast (0.5%) combination (Fig. 1).

2.3. Statistical experimental design

To select the best enzyme concentration and incubation time, a completely randomized factorial design with treatments distributed in a scheme of two enzymes (Ultrazym 100G and Ultrazym 100G + 0.5% Celluclast) was applied; using three incubation times: 30, 75 and 90 min, and three enzyme concentrations: 100, 200 and 300 ppm, in triplicate, at maximum, minimum and medium incubation times and enzyme concentration values. Enzyme concentration and incubation time constituted the independent variables, and juice apparent viscosity, the dependent variable (Reinhardt et al., 2004).

Table 1
Enzymatic pre-treatments with Ultrazym 100G isolatedly, and combined with Celluclast (0.5%) concentrations and incubation times at 30 °C

Assays	1	2	3	4	5
Concentration (ppm)	300	100	300	100	200
Incubation time (min)	30	30	90	90	75

Glucose, fructose and sucrose analyses were carried out, in triplicate, according to a previous experimental design (Carvalho, 2004). All data were treated by analysis of variance (ANOVA) considering the hydrolyzed juice as a standard sample (control). Statistical analyses were conducted with the hydrolyzed juice and MF juice samples, and the hydrolyzed juice and UF juice samples. Multiple comparisons among treatment means were accomplished by the least significance difference (LSD) test at the level of 5% of probability. Statistical analysis was performed using the Statistica software, version 5.1, 98th edition (StatSoft Inc., USA).

2.4. Membrane processes

Pineapple juice was clarified using the PROTOSEP-IV pilot module from Koch (Koch Membrane Systems Inc., Massachusetts, USA). The 20 L feed tank capacity module was equipped with a 0.3 μm pore diameter MF tubular polyethersulfone membrane with filtration area of 0.05 m^2. Trans-membrane pressures (TMP) of 1.5 and 3.0 bar, at room temperature (25 °C ± 2) were applied. For ultra-filtration processes, the same pilot module was used, using a 30–80 KDa (PVdF) tubular UF membrane, filtration area of 0.05 m^2, 1.5 bar, at the same temperature. Another pilot module supplied by DSS - M20 (DSS Filtration Systems Silkborg AS, Silkeborg, Denmark) was also used. Module M20-DSS (plate and frame), with an 8 L feed tank, was equipped for each process with 40 flat sheet polysulfone membranes with MWCO (molecular weight cut-off) of 50 and 100 KDa, respectively. The filtration area was 0.72 m^2, and 6.0 and 7.5 bar TMP were applied. The same plate and frame module was used for MF using a 0.1 and a 0.45 μm pore diameter PS membranes, at 3.5; 4.5 and 5.5 TMP, and 1.5 and 3.0 bar, respectively. All processes were carried out in duplicate and at the same temperature (25 °C ± 2). Since the tubular and plate and frame membrane modules present different geometries and configurations, it was interesting to investigate which one could give the best sugar recoveries. Table 2 summarizes the conditions and some previous results regarding initial volume of hydrolyzed juice, permeates, and hydraulic and permeate fluxes.

2.5. Sucrose, glucose and fructose analyses

Sugar determination was carried out by High Performance Liquid Chromatography from Waters, model W410 (Waters Co., Milford, Massachusetts, USA) with

Table 2
Operating conditions of pineapple juice micro-filtration and ultra-filtration

Membrane	TMP (bar)	Hydraulic flux (L/m^2·h)	TMP (bar)	Permeate flux (L/m^2·h)	Hydrolyzed juice (L)	Permeate volume (L)
PS						
50 KDa	6.0	168.9	6.0	17.4	9.8	5.8
	7.5	223.8	7.5	16.0	9.7	7.2
100 KDa	6.0	580.3	6.0	13.4	10.0	7.8
	7.5	671.8	7.5	19.8	10.0	6.8
0.1 μm	3.5	225.0	3.5	30.6	8.0	6.2
	4.5	275.0	4.5	31.4	7.7	6.0
	5.5	320.0	5.5	20.6	7.3	7.6
0.45 μm	3.0	379.2	3.0	20.0	7.8	5.7
	4.0	491.8	4.0	20.0	7.5	6.1
PVdF						
30–80 KDa	1.5	2427.0	1.5	35.7	18.0	10.5
	2.0	3289.0	–	–	–	–
PES						
0.3 μm	1.5	3109.7	1.5	57.5	18.0	11.2
	2.0	4255.3	3.0	46.8	14.5	9.3

refractive index detector Waters model W410 (Waters Co., Milford, Massachusetts, USA). The analytes were separated on a Waters Sugar Pak ion-exchange column (30 cm by 6.2 mm), in the calcic form; ultra pure de-ionised water as mobile phase was used with a flux of 1 mL/min, according to Mello and Castro (1999).

The sugar analyses were based on gel permeation chromatography (GPC). This specific method was validated for this matrix (pineapple juice) for decreased analysis time and increased sensitivity and repeatability. This method can separate glucose and fructose from the other sugars in the sample with good resolution. The oligosaccharides are separated according to the number of sugar units. Those with different numbers of sugar units are easily separated in the GPC column, but the molecules with exactly the same number of sugar units have the same retention in the column and are not separated. The classic method for sugar analysis by HPLC uses a NH_2 column and acetonitrile/water as mobile phase (Folkes and Crane, 1998). In our method, the use of a GPC column requires a replacement of the mobile phase by water. This change allows the minimisation of the organic solvent toxicity used as mobile phase as well as reduced analysis cost. All sugar analyses were carried out in triplicate.

3. Results and discussion

3.1. Enzymatic hydrolysis

After Ultrazym 100G, and Ultrazym 100G combined with Celluclast at 0.5% treatments, it was observed that the best response in pineapple juice apparent viscosity reduction (27.62%) occurred when Ultrazym 100G (100 ppm), was used alone during 30 min of incubation time (Carvalho et al., 2000). Ultrazym 100G combined with Celluclast (0.5%) promoted an apparent viscosity reduction of 21.11% at the same enzyme concentration and incubation time. Reduced apparent viscosity values (mPa s) of pineapple juice can be observed on Fig. 2. The experiments were carried in triplicate.

3.2. Content of sugars (sucrose, glucose and fructose)

The limitation of the method used for sugar determination is the fact that the oligosaccharides with the same number of sugar units cannot be separated using this type of GPC column. All the disaccharides, such as maltose, sucrose, and lactose, have the same retention time being eluted at the same peak, and thus cannot be separated. In the case of fruit juices, which have just one disaccharide – sucrose, the quantification of three sugars (sucrose, glucose and fructose) can be done without restrictions.

Fig. 3 and Table 3 show the sucrose, glucose and fructose contents after membrane processes. The results revealed significant differences at a 5% level among them.

The sucrose contents decreased in all membrane processes when compared to hydrolyzed pineapple juice (10.24 g/100 mL; SD ± 0.1). The best sucrose recovery was obtained in the permeate juice from the process with 100 KDa PS membranes (74.21%) operating at 7.5 bar (7.60 g/100 mL; SD ± 0.03), and the lowest one (51.17%) with 0.3 μm PES tubular one (5.24 g/100 mL; ±SD 0.1) at 3.0 bar. There were significant sucrose content differences among all samples, with hydrolyzed juice showing the highest content. There was a decline in sucrose content for all permeates from MF processes. But no statistical differences were observed between contents from 0.1 μm to 4.5 bar and 0.45 μm to 4.0 bar MF processes.

The same profile can be seen for UF processes, which also caused a decrease in permeate sucrose contents (Table 3). A statistical difference was observed among all treatments. The permeate juice from process using 30–80 KDa – 1.5 bar membrane showed less sucrose content. According to Youn et al. (2004), this was probably due to the enzyme action and pore size difference between membranes. That is,

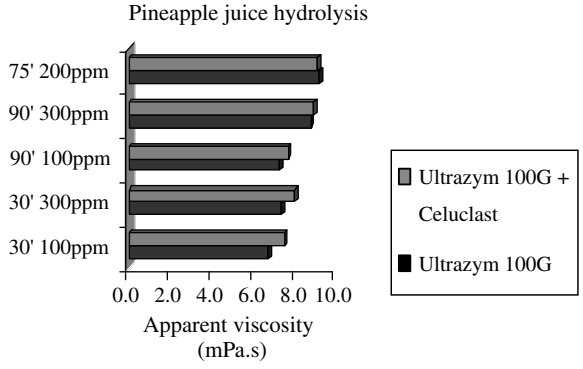

Fig. 2. Apparent viscosity reduction after enzymatic treatments with Ultrazym 100G and combined with Celluclast 0.5%.

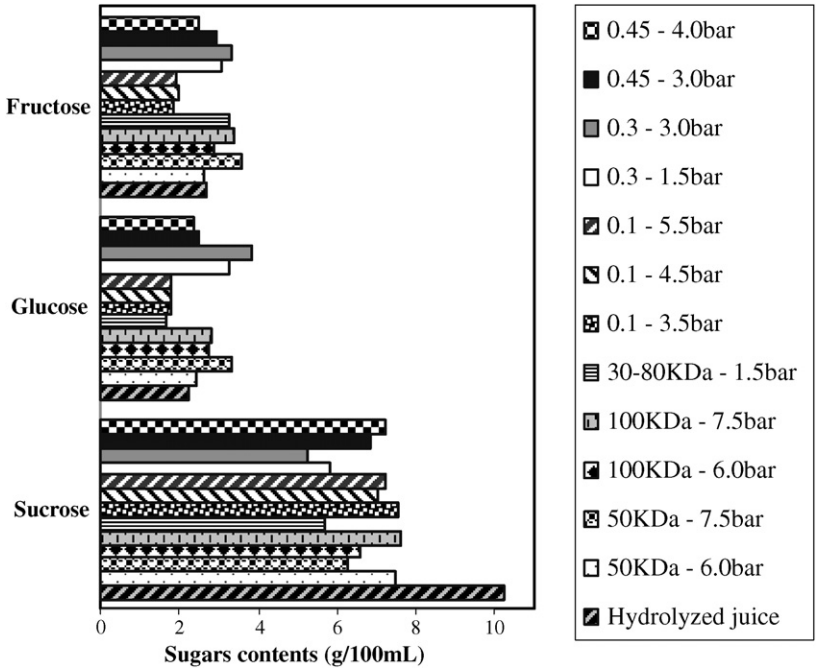

Fig. 3. Sugars contents in hydrolyzed and in clarified pineapple juices. All experiments were carried out in triplicate.

Table 3
Sugars contents in permeate juices from UF and MF processes.

Samples	Sucrose	Glucose % (g/100 mL) SD	Fructose
Hydrolyzed juice	10.24^{a} (±0.1)	2.26^{d} (±0.4)	2.71^{c} (±0.5)
0.1 μm – 3.5 bar	7.54^{b} (±0.02)	1.79^{e} (±0.03)	1.88^{f} (±0.02)
0.1 μm – 4.5 bar	7.06^{c} (±0.03)	1.80^{e} (±0.01)	2.01^{e} (±0.01)
0.1 μm – 5.5 bar	7.21^{d} (±0.1)	1.80^{e} (±0.02)	1.93^{f} (±0.01)
0.3 μm – 1.5 bar	5.81^{f} (±0.06)	3.27^{b} (±0.02)	3.09^{b} (±0.03)
0.3 μm – 3.0 bar	5.24^{g} (±0.1)	3.81^{a} (±0.02)	3.32^{a} (±0.01)
0.45 μm – 3.0 bar	6.82^{e} (±0.1)	2.49^{c} (±0.01)	2.96^{b} (±0.04)
0.45 μm – 4.0 bar	7.25^{c} (±0.1)	2.37^{d} (±0.04)	2.51^{d} (±0.05)
Hydrolyzed juice	10.24^{a} (±0.1)	2.26^{d} (±0.4)	2.71^{e} (±0.5)
100 KDa – 6.0 bar	6.57^{d} (±0.01)	2.78^{c} (±0.02)	2.88^{d} (±0.1)
100 KDa – 7.5 bar	7.60^{b} (±0.03)	2.84^{b} (±0.01)	3.37^{b} (±0.03)
50 KDa – 6.0 bar	7.51^{c} (±0.1)	2.41^{e} (±0.02)	2.65^{e}(±0.03)
50 KDa – 7.5 bar	6.28^{e} (±0.1)	3.33^{a} (±0.01)	3.56^{a} (±0.02)
30–80 KDa – 1.5 bar	5.67^{f} (±0.04)	1.64^{f} (±0.1)	3.27^{c} (±0.03)

Same letters in the same column present no statistical differences for least significance difference (LSD) test ($p < 0.05$).
SD – standard deviation.

enzymes may produce small particles from large polymers, which can penetrate easily through the large pore of the MF membrane but not through the small pore of the UF membrane. A sucrose decrease (1.89–0.83 g/100 mL) was observed as well as glucose (2.84–3.52 g/100 mL) and fructose increases (6.79–7.60 g/100 mL) after apple juice MF (0.1 μm hollow fiber PS membrane), and also in the UF (30 KDa – hollow fiber PS membrane) with glucose increasing from 2.67 to 2.87 g/100 mL, and fructose from 6.36 to 6.83 g/100 mL.

The fructose contents from the MF permeate juices showed the same profile as the glucose contents with 0.3 μm membrane at 1.5 and 3.0 bar, and 0.45 μm membranes at 3.0 bar presenting more fructose than the hydrolyzed juice (Table 3).

With the exception of the permeate juice from the 50 KDa – 6.0 bar process, the fructose contents of permeates from the UF presented a significant difference compared with the hydrolyzed juice.

Fructose was observed to be most preserved in permeate juices clarified with PS 50 KDa – 7.5 bar (3.56 g/mL, SD ± 0.02); PS 100 KDa – 7.5 bar (3.37 g/mL, SD ± 0.03), and PES 0.3 μm – 3.0 bar (3.32 g/mL, SD ± 0.01) membranes.

Similar results were also obtained by Youn et al. (2004), after clarification of apple juice by MF and UF, with fructose increasing from 6.79 (control juice) to 7.60 g/100 mL, and from 6.36 to 6.83 g/100 mL, respectively.

It can be observed that permeates from MF processes using membranes from 0.3 μm to 0.45 μm – 4.0 bar presented the highest glucose contents compared with the hydrolyzed juice, whereas 0.1 μm membrane permeates obtained at all TMP presented the lowest glucose contents. It can be also observed that permeates from 0.3 μm membrane processes at all TMP were higher than hydrolyzed juice. The exception was for the 30–80 KDa membrane process that presented the lowest glucose content (Table 3).

Glucose was more preserved in the permeate juices clarified with PES 0.3 μm – 3.0 bar (3.81 g/mL; SD ± 0.02); PS 50 KDa – 7.5 bar (3.33 g/mL; SD ± 0.01); PES 0.3 μm – 1.5 bar (3.27 g/mL; SD ± 0.02) membranes, confirming data reported by Chao et al. (1992) as well as by Youn

et al. (2004) after apple juice clarification by MF and UF where glucose values ranged from 2.84 (control juice) to 3.52 g/100 mL.

The glucose/fructose ratio for the permeate juices analysed in this work was near 1, maintaining the same relation reported previously (Wallrauch, 1992) for single strength pineapple juice extracted from the bark. However, this relation was 0.50 for 30–80 KDa tubular PVdF membrane process.

Total sugar recoveries with 0.1 μm (3.5; 4.5 and 5.5 bar); 0.3 μm (1.5 and 3.0 bar), and 0.45 μm (1.5 and 3.0 bar) membrane processes were: 73.70%; 71.46% and 71.92%; 80.01%, and 81.32%; 80.67% and 79.75%, respectively.

In the PS membranes, processes with little cut-offs (50 and 100 KDa) total sugar recoveries were higher, with a maximum sugar recovery of 90.79% (100 KDa – 7.5 bar). Chao et al. (1992) obtained superior sugar recoveries (97%) and better permeate flux (24.8 L/m^2 h) in passion fruit juice clarification with 100 KDa PS membranes, operating at 5.0 bar. On the other hand, Youn et al. (2004) did not find significant differences in total sugar contents, after MF and UF of apple juice with 0.1 μm and 30 KDa PS hollow fiber membranes.

The clarification of single strength pineapple juice, at 12 °Brix using a 50 KDa tubular ceramic membrane promoted sucrose and glucose recoveries of 88.23% and 89.06%, respectively (Carvalho et al., 1998), whereas in this work, PS membranes with same cut-off had recoveries of 74.21% and 90.03%. Sugar recoveries were 79.90% with PS membranes and 89.68% with 50 KDa tubular ceramic (α-alumina) membrane. In these authors' study, the 50 KDa tubular ceramic membrane, due to its configuration, promoted a smaller concentration polarization (more turbulence) than the 50 KDa PS flat sheet ones. Also, since non-hydrolyzed pineapple juice was used in the 50 KDa ceramic membrane process, macromolecules with higher molecular weight could have promoted a self-cleaning effect on the tubular membrane surface, minimising concentration polarization and resulting in a best sugar recovery in the permeate juice.

The best permeate fluxes were observed with MF membranes of 0.3 μm (tubular – PES), and 0.1 μm (flat sheet – PS), and the worst ones with 0.45 μm (flat sheet–PS) membranes, 31.37; 57.55 and 20.00 L/m^2 h, respectively (Table 2). We believe that the TMP (0.45 μm – 3.0 and 4.0 bar) applied were higher than necessary causing a fast concentration polarization layer, whose flux became governed by the porosity of the layer, or even by the pore obstruction at the beginning of the processes. On the other hand, membranes with higher permeability foul faster than those with lower permeability (Howe, 2001, 2007).

The maximum permeate fluxes in the UF processes were 17.39 L/m^2 h (50 KDa – 6.0 bar) and 19.77 L/m^2 h (100 KDa – 7.5 bar), respectively. Kim et al. (1995) observed that polysulfone flat sheet membranes promoted the highest flux decline and tended to form concentration polarization layers more densely than PVdF and PES. Riedl et al. (1998) and Gehlert et al. (1998) used 0.1 μm and 100 KDa polysulfone helicoidal (spiral wound) and flat sheet membranes, obtaining better permeate fluxes with helicoidal ones.

On the other hand, UF and MF membranes obtained from different materials (cellulose acetate, polysulfone, polyvinylidene fluoride, polyethersulfone, polycarbonate and ceramic), configurations (flat sheet, tubular and spiral), and module geometries (plate and frame, spiral wound and tubular), influenced the formation of concentration polarization. Normally, flat sheet membranes fouled more rapidly than the hollow fiber ones (Capanelli et al., 1992; Vyas et al., 2000; Curcio et al., 2001; Barros et al., 2003; Hakimzadeh et al., 2006; Howe et al., 2007).

Normally, constituent losses occur in the clarification processes, mainly those of high molecular weight, such as pectin and starch, which are undesirable in both cases since they are responsible for juice turbidity. The monosaccharides and disaccharides can also be reduced since those processes are based on selectivity regarding the membrane pore diameter and particles (Carvalho et al., 2000, 2006).

Sugars, soluble solids, organic acids and minerals suffer a less drastic reduction than other constituents of a higher molecular weight. However, in MF/UF processes, on account of membrane cut-off, configuration, structure, temperature (Wang et al., 2005) as well as occurrence of concentration polarization and fouling, those values may be reduced or even enlarged (Vladisavljevi et al., 2003).

When these problems occur, permeability tends to be led by the solute layer formed on the membrane surface (concentration polarization) and could retain low molecular weight compounds in the retentate.

Itoua-Gassaye et al. (1991) observed that pectin hydrolysis occurring previously to apple juice clarification promoted blocking of the internal membrane pores. In our study, enzymatic hydrolysis may have contributed to particle deposition on the membrane surface, since polarization concentration was observed regardless of cut-off or membrane pore diameter.

According to Youn et al. (2004) polysaccharides, proteins and colloidal materials are present as solid materials in juices, which form crystals with increased concentration or turn into gel, which, in turn, accumulates on the surface to form a secondary membrane as filtration goes on. Solute adsorption inside and/or on the membrane may change the MWCO membranes characteristics leading to increasing membrane resistance.

Membrane configuration, i.e., membrane geometry and the way it is mounted and oriented in relation to the flow of water, is crucial in determining the overall process performance.

The plate and frame membrane module requires the product stream to spread across the entire surface of the individual sheets prior to recirculation. This non-uniform flow path promotes slower flow rates, and tends to accelerate the development of the concentration polarization phenomena (laminar) rather than in the tubular (turbulence

$Re > 4000$) or spiral wound (laminar) membranes, which offer the efficiency of uniform flow through the lumen, allowing industrial scale-up (Curcio et al., 2001). Consequently, it is not directly scalable.

On the other hand, the permeate juices from PS flat sheet membrane module presented higher sucrose recovery values, while these values were higher for glucose and fructose in the permeate juice obtained with 0.3 μm PES tubular membrane.

Although the best total sugar recoveries (13.81 and 13.17 g/100 mL) have been observed in juices clarified with polysulfone membranes (100 KDa – 7.5 bar and 50 KDa – 7.5 bar), the use of 0.3 μm PES membrane (12.37 g/mL), due to its tubular configuration and module geometry, is more attractive and appropriate.

The promotion of a high degree of turbulence, high fluxes, low energetic costs per water volume unit, ease of operation and cleaning, as well as the possibility of scale-up are determining factors for using a tubular membrane module in fruit juice clarification.

On the other hand, the plate and frame membrane module used in this work p had the disadvantage of making it necessary to dismount the equipment for individual cleaning of each membrane after the clarification processes, besides being unfit for industrial scale-up.

4. Conclusions

The sucrose, glucose and fructose permeate juice contents from UF and MF processes differ significantly.

In the 100 and 50 KDa polysulfone membrane processes, total sugar recoveries were higher than in those in which tubular membranes, mainly that of 30–80 KDa (69.55%), were used. The best total sugar recoveries were obtained with 50 KDa membranes at 7.5 bar (90.79%). The higher trans-membrane pressures used in 50 and 100 KDa processes could have influenced the higher total sugar recoveries, although sucrose, glucose and fructose losses occurred after all processes. Enzymatic hydrolysis may have contributed to higher particle retention on the membrane surface or internal pore blocking, since concentration polarisation was observed regardless of membrane cut-off or pore diameter.

Although the best total sugar recoveries have been observed in juices clarified with polysulfone membranes (50 KDa – 7.5 bar), the use of 0.3 μm PES, due to its tubular configuration and module geometry, is more attractive and appropriate. Further studies need to be carried out on 0.1 and 0.3 μm polyethersulfone tubular membrane module, at lower TMPs to observe whether total sugar contents and other constituents may be better preserved.

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